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(54) Granulation of pigments

(57) The present invention provides a process for producing a substantially dry, low dusting, free-flowing granular pigment composition, comprising contacting (a) a fluidised bed of pigment, an additive system which (i) wets out the pigment powder surface sufficiently to allow agglomeration of the pigment particles and (ii) binds granules so formed sufficiently strongly to enable them to resist breakdown during the process and during subsequent handling and

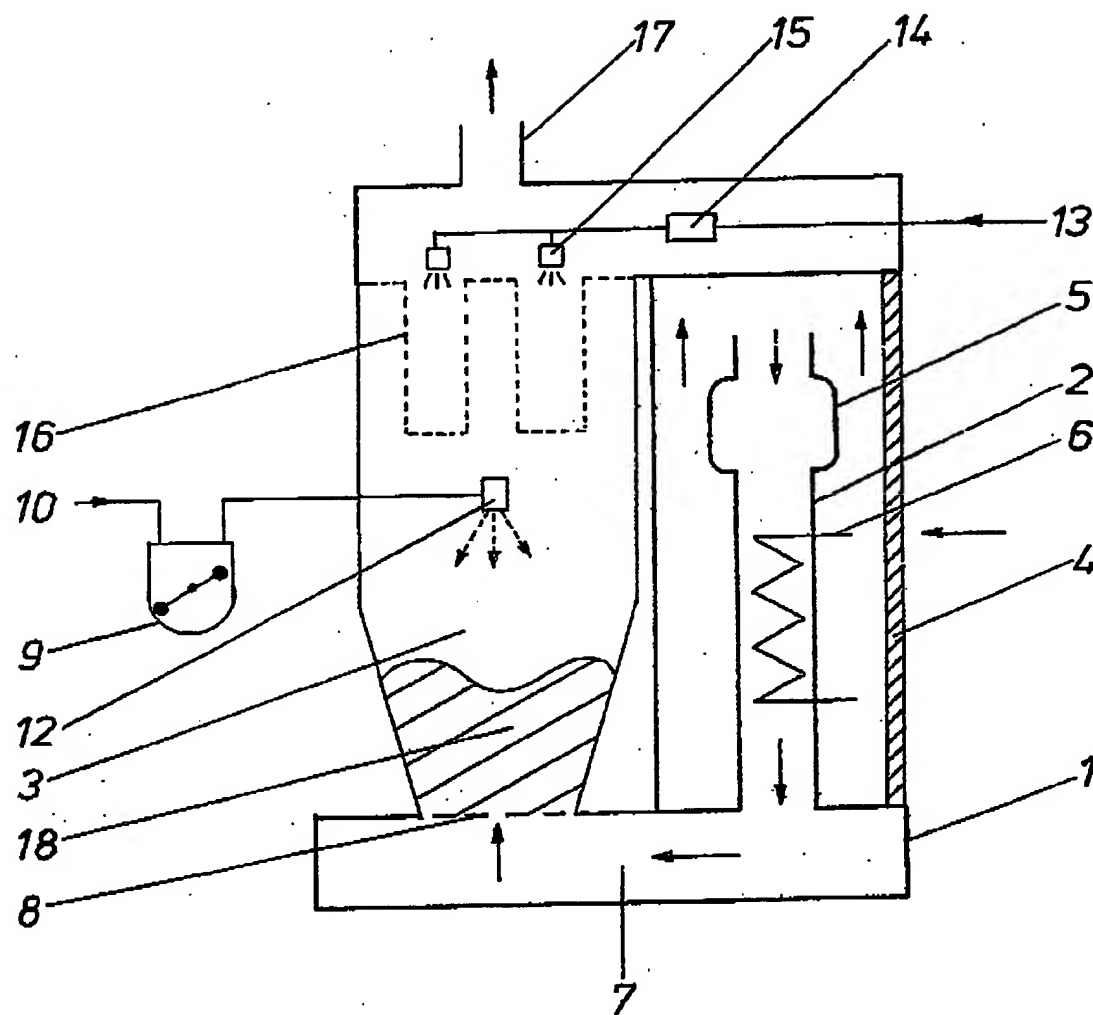
storage but without adversely affecting dispersibility in the final application medium, and water, and (b) removing granules so obtained. Preferred methods include:—

- (i) Mixing the additive system with the fluidised bed of pigment powder and spraying with water; or
(ii) Spraying the additive system and water onto the fluidised bed of pigment.

Suitable additive systems comprise a surface active agent and/or an anionic or nonionic water-soluble polymeric agent.

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SPECIFICATION Granulation of Pigments

The present invention relates to the granulation of pigments in a fluid bed process.

Various fluid bed techniques are already known for the production of materials e.g.

5 pharmaceuticals, agricultural chemicals, dyestuffs, tanning agents, optical brighteners and foodstuffs, in granular form. In a known continuous process, e.g. that described in British Patent Specification No. 1401304, a feed solution, suspension or melt, optionally containing a binder, is sprayed into a drying chamber, through which is blown hot air. Granules are formed and removed continuously; the granules being built up by spray being directed on to previously dried material and subsequent aggregation of such wetted out particles. In this continuous process, drying and granulation thus occur in a single stage. 10

A batch fluid-bed granulation technique has also been described in which a powder-form starting-material is introduced into a fluid bed and sprayed with a solution or emulsion of a binder material. In this way, particles of the active material are coated with the binder, and granules are formed by aggregation of the coated particles. 15

This technique is effected on a batch basis and the starting material has been at least partly dried before introduction into the process.

Neither of these known granulation techniques have thus far been successfully applied to pigment starting materials. The reason, possibly, is that it would have been expected that such 20 relatively severe granulation and/or drying processes, when applied to a pigment, would have had a very disadvantageous effect upon the dispersibility properties of the granular pigment particles rendering them entirely unsuitable for use.

We have now found that by employing a particular additive system, pigments can be successfully granulated in a batch fluid bed process to give products which can be readily incorporated into 25 application media.

Accordingly, the present invention provides a process for producing a substantially dry, low dusting, free-flowing granular pigment composition, comprising (a) contacting a fluidised bed of pigment, an additive system hereinafter described and water, (b) optionally contacting the granules so obtained with the additive system defined in our copending British Patent Application 39682/78 and 30 (c) removing granules so obtained. Preferred methods include:—

(i) Mixing the additive system with the fluidised bed of pigment powder and spraying with water; or

(ii) Spraying the additive system and water onto the fluidised bed of pigment.

By the term "low dusting", we mean a composition having a Dust Index of less than 50, as 35 assessed on the Dust Index as described and explained in British Patent Specification No. 1449283.

A particularly suitable pigment powder starting material is that produced in a conventional dry grinding or sieving process. If desired, prior to use in the present process, such dry-ground pigment powder may be treated to render it more suitable for granulation e.g. by changing its surface properties by chemical means or by grinding less finely—the coarser the powder the less surface to be wetted out 40 and bonded.

Further suitable pigment starting material can be produced from dryers which give pigment directly in powder form.

Such pigment powders may be fully or partially dry and may be low dusting.

The pigment starting material may be an organic pigment or an inorganic pigment. Examples of 45 organic pigments are azo and azomethine pigments or their metal salts or metal complexes, metal phthalocyanines which may be substituted by halogen, polycyclic pigments such as quinacridones, dioxazines, vat dyestuffs, anthraquinones and isoindolinones and salts of basic pigments precipitated with the heteropoly acids of phosphorus, tungsten, molybdenum or copper ferrocyanide. Inorganic pigments which may be used include titanium dioxide, red and yellow iron oxides, carbon black, lead 50 and molybdenum chromes, prussian blues and cadmium reds.

The additive system, used in the process of the present invention may be any material or mixture of materials which:—

i) wets out the pigment powder surface sufficiently to allow agglomeration of pigment particles so wetted out, and 55 ii) binds granules so formed sufficiently strongly to enable them to resist breakdown during the process and during subsequent handling and storage, but without adversely affecting dispersibility in the final application medium.

The additive system should desirably also function to improve the properties of the pigment in its end application.

60 Examples of preferred components of the additive system include surface-active agents of the non-ionic, cationic or anionic type. Surfactants, of course, serve to wet out the pigment particles in the process of the invention and, depending upon the surfactant, the pigment being granulated and the type of final application, the surfactant may also serve as a binding agent and/or a co-additive, functioning to improve the properties of the pigment.

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Typical non-ionic surfactants include those falling within the following sub-groups:—

- a) monoethers of polyglycols with long-chain fatty alcohols. For instance, the condensation product of from 5 to 20 moles of ethylene oxide with a C16—C18 fatty alcohol e.g. cetyl alcohol.
- 5 b) monoesters of polyglycols with long-chain fatty acids; for instance, the condensation products of 5 to 20 moles of ethylene oxide with a fatty acid having 12 to 18 carbon atoms, for example lauric acid or stearic acid. A specific example is polyoxyethylene monolaurate.
- c) monoethers of polyglycols with alkyl-phenols; for instance the reaction product of from 5 to 20 moles of ethylene oxide with a C6—12 alkyl phenol, e.g. nonyl phenol.
- 10 d) N,N-polyethoxylated long-chain fatty amines; for instance the reaction product of from 5 to 50 moles of ethylene oxide with a C12—C18 fatty amine e.g. cocoamine and tallow amine.
- e) N,N-polyethoxylated long-chain fatty acid amides; for instance, the reaction product of from 5 to 50 moles of ethylene oxide with hydrogenated tallow amide.
- f) ester/ethers of polyglycols with cyclic alcohols and fatty acids; specific examples are polyoxyethylene sorbitan oleate or laurate.
- 15 g) condensation products of polyglycols e.g. the condensation product of polyoxypropylene and polyoxyethylene glycols; and
- h) glycols of alkynes e.g. acetylenic glycols.

Typical cationic surface-active agents include primary, secondary and tertiary amines and alkyl propylene diamines. These diamines are typically used either as the C₁—C₄ carboxylic acid salt or as derived quaternary ammonium compounds to give a solution, dispersion or emulsion suitable for spraying.

The anionic surfactants may be alkali metal, ammonium or amine salts (especially volatile amines of low molecular weight such as morpholine or triethylamine) of fatty carboxylic acids and sulphonic acids of fatty amine sulphates. Examples are alkali metal stearates alkali metal alkyl sulphonates e.g. potassium dodecyl sulphonates; alkali metal alkaryl sulphonates e.g. sodium dodecyl benzene sulphonate; fatty acid sarcosinates; sulphonated alkyl esters of long chain fatty acids and alkyl sulphosuccinates; alkali metal salts of polyacrylic acids, sulphonated monoethers of polyglycols with alkylphenols e.g. nonylphenols; and especially the ammonium salt of partly hydrogenated wood rosin.

The surfactant component of the additive system may be used alone, as a mixture of different surfactants or used in conjunction with, or replaced by, a binder and/or an applicational agent known to improve the properties of a pigment, provided that the total additive system meets the functional requirements (i) and (ii) hereinbefore described.

Water soluble polymeric agents are of particular value as binders. They may be anionic or nonionic in nature. Typical products are modified cellulose derivatives such as hydroxyethyl cellulose, hydroxypropyl cellulose, sodium carboxymethyl cellulose, polyvinyl alcohol of varying degrees of hydrolysis from the acetate and polyvinyl pyrrolidone.

Examples of applicational agents known to enhance pigment properties in specific applications (e.g. in plastics, printing inks or paints) are abietic acid and esters thereof; (high molecular weight polyglycols e.g. polyethylene glycol 4000, diglycol monostearate, cellulose acetobutyrate; alkaline earth metal salts of fatty acids having 12 to 20 carbon atoms e.g. stearic acid; fatty acids having 12 to 20 carbon atoms; fatty alcohols; amines having 12 to 20 carbon atoms e.g. stearylamine or rosin oil amine; vinyl chloride polymers; vinyl chloride/vinyl acetate copolymers, polyethylene; polyacrylonitrile or polyterpene resins; dioctyl phthalate, dicyclohexyl phthalate; triglycerides of 12-hydroxystearic acid; maleic and phenolic modified resins; wood resins modified by hydrogenation or polymerisation, and glyceryl esters of modified resins.

Those applicational agents which are water insoluble may be present in the fluidised bed with the pigment or can be added in the spray by formation of a suitable dispersion or emulsion. This dispersion or emulsion may be formed and/or stabilised by selected water soluble agents mentioned before; in such cases the addition of further water soluble agent in the spray may not be necessary.

The choice of the type and relative concentration of the components of the additive system depends on the functional requirements (i) and (ii) hereinbefore described and on such factors as the nature of the application (e.g. ink, plastic or paint), the nature of the pigment surface (e.g. polar/non-polar or hydrophilic/hydrophobic) and on the surface area of the pigment. The nature of the pigment and its method of production are very important in the choice of the additive system. To facilitate the granulation process, the additives previously described may also be added to the pigment at an earlier stage of the manufacturing process e.g. during or after the reaction or during drying or milling. This addition does not enable granulate formation at those stages but can make the subsequent granulation process easier e.g. by requiring lower amounts and/or elimination of one or more components from the additive system to be sprayed on.

The amount of additive system used in the process, expressed as a percentage by weight on the weight of the pigment, can be as high as 100%. However, primarily for reasons of cost, it is preferred to use amounts of additive system within the range of from 0.5 to 20% by weight, on the weight of pigment.

The liquid used to form the solution, emulsion or dispersion of the additive system is preferably

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water, although a minor proportion of organic solvents may be used if desired. The presence of large quantities of an organic solvent is less preferred since, when this is vapourised, leaving the additives deposited on the pigment, the solvent may have to be recovered, thereby increasing the cost and could present a fire/explosion hazard, particularly when the possibility of hybrid mixtures of inflammable vapours and fine dusts are considered.

In cases where the additive system is sprayed onto the bed in conjunction with water, the concentration of the additive system in the solution, emulsion or dispersion may be varied over a wide range provided that the viscosity is appropriate for easy dispensing on to the fluid bed. Concentrations which are too dilute are also disadvantageous in that longer dispensing times, longer cycle times and reduced throughput result. In aqueous systems, the preferred concentration of the additive system is in the range of from 0.5% to 60% by weight, so long as its properties e.g. its viscosity make it suitable for spraying on to the bed.

In one embodiment, the process of the invention is carried out on a batchwise basis, in a machine of the type described in Figure 1 of the accompanying drawing.

In order that the invention may be better understood, and its preferred features explained, a preferred embodiment of the invention will now be described with reference to the accompanying drawing which is a diagrammatic side view, partly in section, of an apparatus for carrying out the process of the invention.

The apparatus comprises a container 1, consisting of an air-heating compartment 2 and, interconnected therewith, a fluid bed compartment 3. The air-heating compartment 2 is equipped with an air filter 4, a suction fan 5 and a heater 6. In an alternative apparatus (not shown), fan 5 is situated in outlet port 17 so that air is drawn rather than forced through the fluid bed. Compartments 2 and 3 are connected by a duct 7 having an air distributor plate 8, forming a partition with the fluid bed compartment 3.

The fluid bed compartment 3 has a spray unit comprising a metering pump 9, with an inlet 10 for a solution, emulsion or dispersion of the additive system and an outlet 11 leading to a nozzle head 12. Above the nozzle head 12 are situated filter bags 16 to retain the material to be granulated within the fluid bed compartment 3. Above the bag filters, there is situated a filter bag cleaning unit comprising an inlet 13, a timer 14 and air jets 15. Alternatively, the bag filters may be cleaned by a shaking device.

At the top of the fluid bed compartment 3 there is an air exhaust port 17. The material to be granulated is placed at the bottom of the fluid bed compartment 3, in the space 18.

The process of the invention is carried out using the apparatus illustrated, as follows:

Dry or semi-dry pigment powder, with or without additives as desired is charged into space 18 of the fluid bed compartment 3. Hot air produced in compartment 2 is blown through the pigment powder to fluidise it. The inlet air temperature at the fluid bed is within the range 50 to 150°C, normally 60 to 120°C; and the outlet air temperature from the bed is in the range 25 to 120°C, depending upon the inlet temperature, the additive system and the rate of dispensation of the latter. The rate of the air flow through the fluid bed is adjusted to give suitable fluidisation of the powder. The rate of the air flow will vary depending upon such factors as machine size, batch size, pigment particle size and density. For a given system it can also be varied during the granulation cycle e.g. as the pigment particle size, shape and density change progressively and thereby change in fluidisation characteristics.

Air is turned on to the filter bag cleaning unit, allowing a periodic air pulse to blow off any pigment adhering to the filter bags 16. Fine dust is therefore continually removed and returned to the fluidised granulation space 18.

Granules are formed by spraying water or a solution, emulsion or suspension of an additive system into the fluidised granulation space 18. The material to be sprayed is metered to the nozzle head 12 whereat it is atomized with or without compressed air depending on nozzle design. Spraying time can vary depending upon the additive system concentration in the spray; the amount of additive system required; the form of the pigment; the rate of air flow; and the temperature. Normally however, the spraying time will be within the range of from 10 minutes to 2 hours.

After granule formation is complete, fluidisation is normally continued for a further period e.g. 1 to 10 minutes, to complete drying to the required degree. This may be effected at a temperature lower than that used in the granulating process to avoid over-heating the product.

Finally, the granules formed are removed from the fluid bed space 18. The products of the process of the present invention are substantially dry (containing not more than 2% by weight of water in most cases), low dusting, easily handleable free-flowing, meterable pigment granules which enable working environments to be cleaner and more free of health hazards.

The following Examples further illustrate the present invention.

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Example 1

Dry pigment powder was charged to the container of the apparatus as shown in the Figure. The process can be summarised as follows:—

- (a) **Process Conditions:**
- Inlet Temperature: 110°C
- Outlet Temperature: 30°C
- Air Flow: 10—15 m³/hour
- Pigment Charge: 250 parts (by weight) of CI Pigment Yellow 13
- Blinder: 500 parts (by weight) of composition:
- 5.0% Atlox 1087 (polyoxyethylene sorbitan oleate)
- 0.25% Klucel M (hydroxypropyl cellulose)
- 94.75% Water
- Spraying Time: 2 hours
- Drying Time: 10 minutes
- (b) **Product Composition**
- 90.5% CI Pigment Yellow 13
- 9.0% Atlox 1087
- 0.5% Klucel M
- (c) **Physical Appearance**
- A low dusting, free flowing granular product having the following size distribution:

<75 μ	75—125 μ	125—180 μ	180—250 μ	250—355 μ	355—500 μ	500—600 μ	600—850 μ	850—1180 μ	1180—2500 μ
1.0%	6.0%	9.5%	19.0%	20.6%	16.6%	8.0%	11.0%	5.5%	2.0%

(Size in Microns)

- (d) **Application Properties**
- The Application properties of the granules and the starting powder were then compared by incorporating equal amounts of pigment in each case into a decorative alkyd paint medium. The resulting paints were then examined and, within practical limits, no differences in applicational properties were observed.

Example 2

- Dry pigment powder was granulated in the apparatus shown in the Figure. The process details were as follows:

- (a) **Process Conditions**
- Inlet Temperature: 110°C
- Outlet Temperature: 30°C
- Air Flow: 10—15 m³/hour
- Pigment Charge: 250 parts (by weight) of CI Pigment Yellow 13
- 500 parts (by weight) of composition
- 4.5% Staybelite Resin
- 0.5% Klucel M
- 2.75% NH₃ solution (SG=0.880)
- 92.25% Water
- Spraying Time: 2 hours
- Drying Time: 10 minutes
- (b) **Product Composition**
- 90.9% CI Pigment Yellow 13
- 8.2% Staybelite Resin (Partly hydrogenated wood rosin)
- 0.9% Klucel M
- (c) **Physical Appearance**
- A low dusting, free flowing granular product having the following size distribution:

<75 μ	75—125 μ	125—180 μ	180—250 μ	250—355 μ	355—500 μ	500—600 μ	600—850 μ	850—1180 μ	1180—2500 μ
1.0%	10.0%	12.5%	20.0%	22.9%	18.0%	8.0%	6.5%	1.5%	—

(Size in Microns)

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(d) Application Properties

The application properties of the granules and the starting powder were then compared by incorporating equal amounts of *pigment* into lithographic ink medium. The resulting inks were then examined and, within practical limits, no differences in applicational properties were observed.

5 Example 3

Dry pigment powder was charged to the container of the apparatus shown in the Figure. The process details are as follows:

(a) Process Conditions:

10	Inlet Temperature:	110°C	5
	Outlet Temperature:	30°C	10
	Air Flow:	10—15 m ³ /Sec.	
	Pigment Charge:	250 parts (by weight) of CI Pigment Yellow 13	
	Binder:	500 parts (by weight) of composition	
		5% Lissapol NX (ethoxylated nonyl phenol)	
15		95% Water	15
	Spraying Time:	2 hours	
	Drying Time:	10 minutes.	

(b) Pigment Composition:

20	91% CI Pigment Yellow 13	20
	9% Lissapol NX	

(c) Physical Appearance

A low dusting, free flowing granular product having the following size distribution:

0—75 μ	75—125 μ	125—180 μ	180—250 μ	250—355 μ	355—500 μ	500—600 μ	600—850 μ	850—1180 μ	1180—2500 μ
0.5%	1.5%	3.0%	7.5%	12.5%	19.0%	16.0%	24.0%	15.0%	1.0%

(Size in Microns)

(d) Application Properties

25 The application properties of the granules and the starting powder were then compared by incorporating equal amounts of *pigment* into an acrylic ink medium. The resulting inks were then examined and, within practical limits, no differences in applicational properties were observed. 25

Example 4

30 Dry pigment powder was charged to the container shown in the Figure. The process details are as follows: 30

(a) Process Conditions

35	Inlet Temperature:	110°C	
	Outlet Temperature:	28°C	
	Air Flow:	20—30 m ³ /hour	
	Pigment Charge:	250 parts (by weight) of CI Pigment Yellow 13	35
	Binder:	250 parts (by weight) of composition:	
		10.0% Atlox 1087	
		0.5% Klucel M	
		89.5% Water	
40	Spraying Time:	30 minutes	40
	Drying Time:	10 minutes	

(b) Production Composition

45	90.5% CI Pigment Yellow 13	
	9.0% Atlox 1087	
	0.5% Klucel M	45

(c) Physical Appearance

A low dusting, free flowing granular product having the following size distribution:

0—75 μ	75—125 μ	125—180 μ	180—250 μ	250—355 μ	355—500 μ	500—600 μ	600—850 μ	850—1180 μ	1180—2500 μ
10.5%	8.5%	7.5%	10.5%	13.0%	13.0%	10.5%	14.0%	8.5%	4.0%

(Size in Microns)

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(d) Application Properties

The application properties of the granules and starting powder were then compared by incorporation of equal amounts of *pigment* into a decorative alkyd paint medium. The resulting paints were then examined and, within practical limits, no difference in applicational properties were observed.

Example 5

Dry pigment powder was charged to the apparatus shown in the Figure. The process details were as follows:

(a) Process Conditions

Inlet Temperature: 110°C
 Outlet Temperature: 40°C
 Air Flow: 20—30 m³/hour
 Pigment Charge: 250 parts (by weight) of CI Pigment Blue 15.3
 Binder: 500 parts (by weight) of composition:
 1.5% Atlox 1087
 0.5% Klucel M
 0.5% Lissapol NX (Ethoxylated nonyl phenol)
 97.5% Water

Spraying Time: 2.5 hours
 Drying Time: 10 minutes

(b) Product Composition

95.1% CI Pigment Blue 15.3
 2.9% Atlox 1087
 1.0% Klucel M
 1.0% Lissapol NX

(c) Physical Appearance

A low dusting, free flowing granular product having the following size distribution:

0—75 μ	75—125 μ	125—180 μ	180—250 μ	250—355 μ	355—500 μ	500—600 μ	600—850 μ	850—1180 μ	1180—2500 μ
2.6%	4.5%	4.6%	8.0%	9.0%	2.6%	2.9%	8.0%	18.0%	40.5%

(All sizes in Microns)

(d) Application Properties

The application properties of the granules and the starting powder were then compared by incorporating equal amounts of *pigment* into an industrial paint (Alkyd Melamine) medium. The resulting paints were examined and, within practical limits, no differences in applicational properties were observed.

Example 6

Dry pigment powder was charged to the apparatus shown in the Figure. The process details are as follows:

(a) Process Conditions

Inlet Temperature: 110°C
 Outlet Temperature: 30°C
 Air Flow: 10—15 m³/hour
 Pigment Charge: 250 parts (by weight) of CI Pigment Red 57.1
 Binder: 500 parts (by weight) of composition:
 1.75% Atlox 1087
 0.25% Klucel M
 0.5% Solumin F10S (Na salt of sulphated nonlyphenoxy polyoxyethane)
 97.5% Water

Spraying Time: 2 hours
 Drying Time: 15 minutes

(b) Product Composition

95.2% CI Pigment Red 57.1
 3.3% Atlox 1087
 0.5% Klucel M
 1.0% Solumin F10S

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(c) Physical Appearance

A low dusting, free flowing granular product having the following size distribution:—

0—75 μ	75—125 μ	125—180 μ	180—250 μ	250—355 μ	355—500 μ	500—600 μ	600—850 μ	850—1180 μ	1180—2500 μ
1.25%	11.0%	17.0%	27.25%	28.5%	7.5%	4.0%	2.5%	—	—

(Size in Microns)

(d) Application Properties

- 5 The application properties of the granules and starting powder were then compared by incorporating equal amounts of *pigment* into an industrial paint (Alkyd Melamine) medium. The resulting paints were then examined and, within practical limits, no differences in application properties were observed. 5

- 10 **Example 7**
250 parts (by weight) of CI pigment Yellow 13 are charged to the container (See Figure). The pigment powder is then fluidised with hot air ($100^{\circ}\text{C} \pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 250 parts (by weight) of binder of composition: 10

- 15 5.0% Ethoxylated fatty alcohol (Emulan OG)
0.1% Hydroxy propyl cellulose (Klucel M)
5.0% Hydrogenated wood rosin (Staybelite Resin)
3.0% NH_3 solution ($\text{Sg}=0.880$)
86.9% Water 15

are then sprayed onto the fluidised bed of pigment uniformly over a period of 1 hour. Fluidisation is continued for a further 5 minutes. The resulting granules, of composition (by weight).

- 20 90.0% CI Pigment Yellow 13
4.5% Ethoxylated fatty alcohol
0.2% Hydroxy propyl cellulose
4.5% Hydrogenated wood rosin 20

- 25 had a size distribution similar to that obtained in Example 4. The granules and Original powder were then compared by incorporating equal amounts of *pigment* into a nitrocellulose ink. The resulting inks were then examined and, within practical limits, no differences in application properties were observed. 25

- 30 **Example 8**
250 parts (by weight) of CI Pigment Yellow 13 are charged to the container (See Figure). The pigment powder is then fluidised with hot air ($100^{\circ}\text{C} \pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 850 parts (by weight) of binder of composition: 30

- 35 1.0% Na salt of sulphonated polyoxyalkylene condensate (Solumin Fios at 100%)
6.5% Potassium salt of polymethacrylic acid (Vinapol 1640)
6.7% Glycerol ester of hydrogenated wood rosin (Bresinol 303 at 100%)
91.8% Water 35

are then sprayed onto the fluidised bed of pigment uniformly over a period of 1 1/4 hours. Fluidisation is continued for a further 10 minutes. The resulting granules, of composition (by weight).

- 40 90.1% CI Pigment Yellow 13
1.8% Na salt of sulphonated polyoxyalkylene condensate
0.9% K salt of polymethacrylic acid
7.2% Glycerol ester of hydrogenated wood rosin. 40

- 45 had a size distribution similar to that obtained in Example 2. The granules and Original powder were then compared by incorporating equal amounts of *pigment* into an industrial paint (Alkyd Melamine) medium. The resulting paints were then examined and, within limits, no differences in application properties were observed. 45

- Example 9**
250 parts (by weight) of CI Pigment Yellow 13 are charged to the container (See Figure). The

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pigment powder is then fluidised with hot air ($100^{\circ}\pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 500 parts (by weight) of binder composition:

	2.0% Ethoxylated nonyl phenol (Lissapol NX)	
	0.3% Hydroxypropyl cellulose (Klucel M)	
5	3.0% Maleic modified resin (Pentalyn 255)	5
	1.8% NH_3 Solution ($\text{SG}=0.830$)	
	92.9% Water	

are then sprayed onto the fluidised bed of pigment uniformly over a period of 2 hours. The resulting granules, of composition (by weight)

10	90.5% CI Pigment Yellow 13	10
	3.6% Ethoxylated nonyl phenol	
	0.5% Hydroxypropyl cellulose	
	5.4% Maleic modified resin	

had a size distribution similar to that obtained in Example 3.

15	The granules and Original powder were then compared by incorporating equal amounts of pigment into a nitrocellulose ink.	15
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The resulting inks were then examined and, within practical limits, no differences in application properties were observed.

Example 10

20	250 parts (by weight) of CI Pigment Yellow 13 are charged to the container (See Figure). The pigment powder is then fluidised with hot air ($100^{\circ}\text{C}\pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 300 parts (by weight) of binder of composition:	20
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	3.6% C_{18} Fatty amine (Crodamine IT)	
	0.3% Polyvinyl alcohol (Elvanol 50.42)	
25	1.5% Ethoxylated fatty amine (Catafor 09)	25
	94.7% Water	

are then sprayed onto the fluidised bed of pigment uniformly over a period of 1 hour. Fluidisation is continued for a further 5 minutes. The resulting granules, of composition (by weight).

30	90.5% CI Pigment Yellow 13	30
	6.3% C_{18} Fatty amine	
	0.5% Polyvinyl alcohol	
	2.7% Ethoxylated fatty amine	

had a size distribution similar to that obtained in Example 4.

35	The granules and Original powder were then compared by incorporating equal amounts of pigment into a nitrocellulose ink.	35
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The resulting inks were then examined and, within practical limits, no differences in application properties were observed.

Example 11

40	250 parts (by weight) of CI Pigment Yellow 109 are charged to the container (See Figure). The pigment powder is then fluidised with hot air ($100^{\circ}\text{C}\pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 500 parts (by weight) of binder of composition:	40
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	0.25% Hydroxy ethyl cellulose (Natrosol 250 HR)	
	4.00% Hydrogenated wood rosin (Staybelite Resin)	
	2.45% NH_3 Solution ($\text{SG}=0.880$)	
45	1.00% Sodium dioctyl sulphosuccinate (Anonaid THQ 100%)	45
	92.30% Water	

are then sprayed onto the fluidised bed of pigment uniformly over a period of 2 hours. Fluidisation is continued for a further 10 minutes. The resulting granules of composition (by weight)

50	90.6% CI Pigment Yellow 109	50
	7.2% Hydrogenated wood rosin	
	0.6% Hydroxyethyl cellulose	
	1.6% sodium dioctyl sulphosuccinate	

had a Dust Index 19 (Original powder 23) and a size distribution similar to that obtained in Example 5.

9

The granules and Original powder were then compared by incorporating equal amounts of *pigment* into an industrial paint medium (Alkyd Melamine).

The resulting paints were then examined and, within practical limits, no differences in application properties were observed.

5 Example 12

250 parts (by weight) of CI Pigment Yellow 109 are charged to the container (See Figure). The pigment powder is then fluidised with hot air ($100^{\circ}\text{C} \pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 350 parts (by weight) of binder of composition:

- | | | |
|-------|--|----|
| 2.9% | Polyoxypropylene (Polyoxyethylene copolymer (MW 8500) (Suronic E800) | 5 |
| 0.4% | Hydroxypropyl cellulose (Klucel M) | |
| 4.3% | Unsaturated C_{18} fatty alcohol (Oleyl alcohol) | 10 |
| 92.9% | Water | |

are then sprayed onto the fluidised bed of pigment uniformly over a period of 1.25 hours. Fluidisation is continued for a further 10 minutes. The resulting granules, of composition (by weight):

- | | | |
|-------|--|----|
| 90.5% | CI Pigment Yellow 109 | 15 |
| 3.6% | Polyoxypropylene/polyoxyethylene copolymer | |
| 0.5% | Hydroxypropyl cellulose | |
| 5.4% | Unsaturated C_{18} fatty alcohol | |

- 20 had a Dust Index of 6 (Original powder 23) had a size distribution similar to that obtained in Example 5. The granules and Original powder were then compared by incorporating equal amounts of *pigment* into an industrial paint medium (Alkyd melamine).

The resulting paints were then examined and, within practical limits, no differences in application properties were observed.

Example 13

- 25 parts (by weight) of CI Pigment Yellow 109 are charged to the container (See Figure). The pigment powder is then fluidised with hot air ($100^{\circ}\text{C} \pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 250 parts (by weight) of binder of composition:

- | | | |
|--------|---|----|
| 3.0% | Sodium lauroyl sarcosinate (Sarkosyl NL) at | |
| 0.25% | Hydroxy ethyl cellulose (Natrosol 250 HR) | |
| 7.0% | Triglyceride of 12 hydroxy stearic acid (Synthaway) | 30 |
| 89.75% | Water | |

are then sprayed onto the fluidised bed of pigment uniformly over a period of 1 hour. Fluidisation is continued for a further 5 minutes. The resulting granules, of composition (by weight):

- | | | |
|-------|---|----|
| 90.7% | CI Pigment Yellow 109 | 35 |
| 2.7% | Sodium Lauroyl sarcosinate | |
| 0.2% | Hydroxy ethyl cellulose | |
| 8.4% | Triglyceride of 12-hydroxy stearic acid | |

- 40 had a Dust Index of 13 (Original powder 23) and had a size distribution similar to that obtained in Example 3. The granules and Original powder were then compared by incorporating equal amounts of *pigment* into a plastified PVC. The resulting PVC hides were then examined and, within practical limits, no differences in application properties were observed.

Example 14

- 45 250 parts (by weight) of CI Pigment Yellow 62.1 are charged to the container (See Figure). The pigment powder is then fluidised with hot air ($100^{\circ}\text{C} \pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30° . 200 parts (by weight) of binder of composition:

- | | | |
|-------|---|--|
| 3.8% | Sodium dioctyl sulphosuccinate (Aronaid TH at 100%) | |
| 1.3% | Polyvinyl pyrrolidone (Aldacol Q 100%) | |
| 8.0% | Dioctyl phthalate (Reomol D79P) | |
| 80.1% | Water | |

- 50 are then sprayed onto the fluidised bed of pigment uniformly over a period of 3/4 hour. Fluidisation is continued for a further 10 minutes. The resulting granules, of composition (by weight):

10

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90.1% CI Pigment Yellow 62.1
2.7% Sodium dioctyl sulphosuccinate
0.9% Polyvinyl pyrrolidone
8.3% Dioctyl phthalate

- 5 had a Dust Index of 18 (Original powder 30) had a size distribution similar to that obtained in Example 6. The granules and Original powder were then compared by incorporating equal amounts of *pigment* into plastisolised PVC.

The resulting PVC hides were then examined and, within practical limits, no differences in application properties were observed.

10 Example 15

250 parts (by weight) of CI Pigment Yellow 68.1 are charged to the container (See Figure). The pigment powder is then fluidised with hot air ($100^{\circ}\text{C} \pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 350 parts (by weight) of binder of composition:

- 15 8.0% Sorbitan tristearate (Span 65)
0.7% Hydroxy propyl cellulose (Klucel M)
2.1% Cyclohexyl phthalate (Howflex CP)
92.2% Water

are then sprayed onto the fluidised bed of pigment uniformly over a period of 1 1/2 hours. Fluidisation is continued for a further 5 minutes. The resulting granules, of composition (by weight)

- 20 90.1% CI Pigment Yellow 62.1
8.3% Sorbitan tristearate
0.9% Hydroxy propylcellulose
2.7% Cyclohexyl phthalate

- 25 had a size distribution similar to that obtained in Example 2. The granules and Original powder were then compared by incorporating equal amounts of *pigment* into a decorative alkyd paint medium.

The resulting paints were then examined and, within practical limits, no differences in application properties were observed.

Example 16

- 30 250 parts (by weight) of CI Pigment Yellow 62.1 are charged to the container (See Figure). The pigment powder is then fluidised with hot air ($100^{\circ}\text{C} \pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 250 parts (by weight) of binder of composition:

- 35 4.0% Phosphate ester of a polyoxyalkylene condensate (Solumin PFN 60)
0.5% Polyvinylpyrrolidone (Aldacol at 100%)
6.0% Saturated C_{18} fatty acid (Isostearic acid)
89.5% Water

are then sprayed onto the fluidised bed of pigment uniformly over a period of 2 hours. Fluidisation is continued for a further 5 minutes. The resulting granules, of composition (by weight).

- 40 90.5% CI Pigment Yellow 62.1
3.6% Phosphate ester of a polyoxyalkylene condensate
0.5% Polyvinyl pyrrolidone
5.4% Saturated C_{18} fatty acid

had a Dust Index of 14 (Original powder 30) and had a size distribution similar to that obtained in Example 6. The granules and Original powder were then compared by incorporating equal amounts of *pigment* into a decorative, alkyd paint medium.

- 45 The resulting paints were then examined and, within practical limits, no difference in application properties were observed.

Example 17

- 50 260 parts (by weight) of CI Pigment Yellow 13 previously treated with 10 parts (by weight) of abietylamine (Rosin Amine D) are charged to the container (See Figure). The pigment powder is then fluidised with hot air ($100^{\circ}\text{C} \pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 350 parts (by weight) of binder of composition:

11

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- 0.7% Sodium N-methyl n-oleyl laurate (Adinol at 100%)
 0.4% Sodium Carboxy methyl cellulose
 3.6% Maleic modified resin (Pentalyn 255)
 2.2% NH_3 Solution (SG=0.880)
 93.1% Water
- are then sprayed onto the fluidised bed of pigment uniformly over a period of hours. Fluidisation is continued for a further minutes. The resulting granules, of composition (by weight):
- 90.4% CI Pigment Yellow 13
 3.6% Abietylamine
 4.6% Maleic modified Resin
 0.9% Sodium-N-methyl-N-oleyl laurate
 0.5% Sodium carboxy methyl cellulose
- had a size distribution similar to that obtained in Example 4
 The granules and Original powder were then compared by incorporating equal amounts of pigment into an industrial paint medium (alkyd melamine).
 The resulting paints were then examined and, within practical limits, no differences in application properties were observed.
- Example 18
 250 parts (by weight) of CI Pigment Yellow 13 previously treated with 10 parts (by weight) of abietylamine (Rosin Amine D) are charged to the container (See Figure). The pigment powder is then fluidised with hot air ($100^\circ\pm 10^\circ\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 250 parts (by weight) of binder of composition:
- 0.5% Hydroxypropylcellulose (Alucel M)
 6.0% $\text{C}_6\text{---C}_{18}$ Fatty amine acetate (Armac C)
 4.0% Unsaturated C_{18} fatty alcohol (Oleyl alcohol)
 89.5% Water
- are then sprayed onto the fluidised bed of pigment uniformly over a period of 2 hours. Fluidisation is continued for a further minutes. The resulting granules, of composition (by weight):
- 87.5% CI Pigment Yellow 13
 3.5% Abietylamine
 5.2% $\text{C}_6\text{---C}_{18}$ fatty amine acetate
 3.5% Unsaturated C_{18} fatty alcohol
 0.4% Hydroxy propyl cellulose
- had a Dust Index of 30 (Original powder 78) and had a size distribution similar to that obtained in Example 3. The granules and Original powder were then compared by incorporating equal amounts of pigment into an industrial paint medium (alkyd melamine).
 The resulting paints were then examined and, within practical limits, no differences in application properties were observed.
- Example 19
 260 parts (by weight) of CI Pigment Yellow 13 previously treated with 10 parts (by weight) of abietylamine (Rosin Amine D) and 15 parts (by weight) of sodium alkyl naphthalene sulphonate (Altamol NNO) are charged to the container (See Figure). The pigment powder is then fluidised with hot air ($100^\circ\text{C}\pm 10^\circ\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 250 parts (by weight) of binder of composition:
- 0.5% Hydroxy ethyl cellulose (natrosol 250 HR)
 89.5% Water
- are then sprayed onto the fluidised bed of pigment uniformly over a period of 1 1/2 hours. Fluidisation is continued for a further 10 minutes. The resulting granules, of composition (by weight):
- 90.5% CI Pigment Yellow 13
 3.6% Abietylamine
 5.4% Sodium alkyl naphthalene sulphonate
 0.5% Hydroxy ethyl cellulose

had a size distribution similar to that obtained in Example 2.

12

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The granules and Original powder were then compared by incorporating equal amounts of pigment into an industrial paint medium (alkyd melamine).
The resulting paints were then examined, and, within practical limits, no differences in application properties were observed.

5 Example 20

250 parts (by weight) of CI Pigment Yellow 129 are charged to the container (See Figure).
The pigment powder is then fluidised with hot air ($100^{\circ}\text{C} \pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 500 parts (by weight) of binder of composition:

- | | | |
|----|---|----|
| 10 | 0.5% Klucel 'M' (Hydroxy propyl cellulose) | 5 |
| | 2.5% Duomac 'T' (Acetate of tallow propylene diamine) | |
| | 2.5% Olayl alcohol | |
| | 94.5% Water | 10 |

are then sprayed onto the fluidised bed of pigment uniformly over a period of 2 hours. Fluidisation is continued for a further 10 minutes. The resulting granules, of composition (by weight):

- | | | |
|----|-----------------------------|----|
| 15 | 90.1% CI Pigment Yellow 129 | 15 |
| | 0.9% Klucel 'M' | |
| | 4.5% Duomac 'T' | |
| | 4.5% Olayl alcohol | |

had a size distribution similar to that obtained in Example 4.

- | | | |
|----|--|----|
| 20 | The granules and Original powder were then compared by incorporating equal amounts of pigment into an industrial paint medium (Alkyd melamine).
The resulting paints were then examined and, within practical limits, no differences in application properties were observed. | 20 |
|----|--|----|

Example 21

- | | | |
|----|---|----|
| 25 | parts (by weight) of CI Pigment Yellow 129 are charged to the container (See Figure).
The pigment powder is then fluidised with hot air ($100^{\circ}\text{C} \pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 350 parts (by weight) of binder of composition: | 25 |
|----|---|----|

- | | | |
|----|--|----|
| 30 | 0.4% Natrosol 250 HR (Hydroxy ethyl cellulose) | 30 |
| | 5.0% Staybelite resin (Hydrogenated wood rosin) | |
| | 2.1% Supronic E800 (Polyoxypropylene/polyoxyethylene copolymer EMW 8500) | |
| | 3.0% NH_3 solution (S6 20.550) | |
| | 89.5% Water | |

are then sprayed onto the fluidised bed of pigment uniformly over a period of 1 1/2 hours. Fluidisation is continued for a further 5 minutes. The resulting granules, of composition (by weight):

- | | | |
|----|-----------------------------|----|
| 35 | 90.5% CI Pigment Yellow 129 | 35 |
| | 0.5% Natrosol 250 HR | |
| | 6.3% Staybelite resin | |
| | 2.7% Supronic E800 | |

- | | | |
|----|--|----|
| 40 | had a Dust Index of 19 (Original powder 66) and a size distribution similar to that obtained in Example 3. | 40 |
|----|--|----|

The granules and Original powder were then compared by incorporating equal amounts of pigment into an industrial paint medium (Alkyd melamine).

The resulting paints were then examined and, within practical limits, no differences in application properties were observed.

45 Example 22

293 parts (by weight) of CI Pigment Yellow 1 powder of which 30.6 parts (by weight) are water and 12.4 parts Solumin FIOS at 100% (sodium salt of polyoxyalkylene condensate) are charged to the container (see Figure). The pigment powder is then fluidised with hot air ($100^{\circ}\text{C} \pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 200 parts (by weight) of binder of composition:

- | | | |
|----|---|----|
| 50 | 0.8% Natrosol 250 HR (Hydroxy ethyl cellulose) | 50 |
| | 6.3% Dresinol 303 at 100% (Glycerol ester of hydrogenated wood rosin) | |
| | 93.1% Water | |

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are then sprayed onto the fluidised bed of pigment uniformly over a period of 1 1/2 hours. Fluidisation is continued for a further 5 minutes. The resulting granules, of composition (by weight):

- 5 90.4% CI Pigment Yellow 1
 0.4% Natrosol 250 HR
 4.6% Glycerol ester of hydrogenated wood rosin
 4.6% Solumin Fios at 100% 5

had a Dust Index of 6 (Original powder 78) and a size distribution similar to that obtained in Example 3. The granules and Original powder were then compared by incorporating equal amounts of pigment into a nitrocellulose ink.

- 10 The resulting inks were then examined and the ink prepared from the granules had improved strength and gloss. 10

Example 23

- 16 293 parts (by weight) of CI Pigment Yellow 1 powder of which 30.6 parts (by weight) are water and 12.4 parts by weight are Solumin FIOS as 100% sodium salt of polyoxyalkylene (condensate) are charged to the container (See Figure). The pigment powder is then fluidised with hot air (100°C±10°C) and the outlet temperature allowed to rise to approx. 30°C. 260 parts (by weight) of binder of composition: 15

- 20 0.5% Klucel M (Hydroxy propyl cellulose)
 5.0% Staybelite Resin (Hydrogenated wood rosin)
 3.0% NH₃ solution (SG=0.880)
 91.5% Water 20

are then sprayed onto the fluidised bed of pigment uniformly over a period of 1 hour. Fluidisation is continued for a further 10 minutes. The resulting granules, of composition (by weight):

- 25 90.4% CI Pigment Yellow 1
 0.5% Klucel M
 4.6% Staybelite Resin
 4.5% Solumin FIOS 25

had a Dust Index of 4 (Original powder 78) and a size distribution similar to that obtained in Example 6. The granules and Original powder were then compared incorporating equal amounts of pigment into a nitrocellulose ink.

- 30 The resulting inks were then examined and, with practical limits, no differences in application properties were observed. 30

Example 24

- 35 250 parts (by weight) of CI Pigment Yellow 93 are charged to the container (See Figure). The pigment powder is then fluidised with hot air (100°C±10°C) and the outlet temperature allowed to rise to approx. 30°C. 300 parts (by weight) of binder of composition: 35

- 0.4% Natrosol 250 HR (Hydroxy ethyl cellulose)
 5.8% Dresinol 303 at 100% (Glycerol ester of hydrogenated wood rosin)
 2.5% Solumin Fios at 100% (Sodium salt of polyoxyethylene condensate)

- 40 are then sprayed onto the fluidised bed of pigment uniformly over a period of 1 1/2 hours. Fluidisation is continued for a further 10 minutes. The resulting granules, of composition (by weight): 40

- 45 90.5% CI Pigment Yellow 93
 0.5% Natrosol 250 HR
 6.3% Dresinol 303 at 100%
 2.7% Solumin Fios at 100% 45

had a size distribution similar to that obtained in Example 3.

The granules and Original powder were then compared by incorporating equal amounts of pigment into an industrial paint (alkyd melamine) medium.

- 50 The resulting paints were then examined and, within practical limits, no differences in application properties were observed. 50

Example 25

250 parts (by weight) of CI Pigment Yellow 93 are charged to the container (See Figure). The

14

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pigment powder is then fluidised with hot air ($100^{\circ}\text{C} \pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 300 parts (by weight) of binder of composition:

- 5 0.4% Klucel M (Hydroxy propylcellulose)
 5.8% Staybelite resin (Hydrogenated wood rosin)
 3.5% NH_3 solution (SG=0.880)
 2.5% Manoxol OT at 100% (Sodium dioctyl sulphosuccinate)
 87.8% Water

5.

are then sprayed onto the fluidised bed of pigment uniformly over a period of 1 1/2 hours. Fluidisation is continued for a further 10 minutes. The resulting granules, of composition (by weight):

- 10 90.5% of CI Pigment Yellow 93
 0.5% Klucel M
 6.3% Staybelite Resin
 2.7% Manoxol OT at 100%

10

had a size distribution similar to that obtained in Example 5.

- 15 The granules and Original powder were then compared by incorporating equal amounts of pigment into an industrial paint (alkyd melamine) medium.

15

The resulting paints were then examined and, within practical limits, no difference in application properties were observed.

Example 26

- 20 250 parts (by weight) of CI Pigment Red 48.2 are charged to the container (See Figure). The pigment powder is then fluidised with hot air ($100^{\circ}\text{C} \pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 300 parts (by weight) of binder of composition:

20

- 25 0.4% Klucel M (Hydroxy propyl cellulose)
 2.5% Lissapol NX (Ethoxylated nonyl phenol)
 5.8% Staybelite Resin (Hydrogenated wood rosin)
 3.5% NH_3 solution (SG=0.880)
 87.8% Water

25

are then sprayed onto the fluidised bed of pigment uniformly over a period of 1 1/4 hours. Fluidisation is continued for a further 10 minutes. The resulting granules, of composition (by weight):

- 30 90.5% CI Pigment Red 48.2
 0.5% Klucel M
 6.3% Staybelite Resin
 2.7% Lissapol NX

30

- 35 had a Dust Index of 6 (Original powder 40) and a size distribution similar to that obtained in Example 3.

35

The granules and Original powder were then compared by incorporating equal amounts of pigment into an industrial paint medium (alkyd melamine).

The resulting paints were then examined and, within practical limits, no differences in application properties were observed.

Example 27

- 40 250 parts (by weight) of CI Pigment Red 88 are charged to the container (See Figure). The pigment powder is then fluidised with hot air ($100^{\circ}\text{C} \pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 250 parts (by weight) of binder of composition:

40

- 45 0.5% Klucel M (Hydroxy propyl cellulose)
 7.0% Oleyl alcohol
 3.0% Manoxol OT at 100% (Sodium dioctylsulphosuccinate)
 89.5% Water

45

are then sprayed onto the fluidised bed of pigment uniformly over a period of 1 1/2 hours. Fluidisation is continued for a further 5 minutes. The resulting granules, of composition (by weight):

- 50 90.5% CI Pigment Red 88
 0.5% Klucel M
 6.3% Oleyl alcohol
 2.7% Manoxol OT at 100%

50

had a size distribution similar to that obtained in Example 4.

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15

The granules and Original powder were then compared by incorporating equal amounts of pigment into an industrial paint (alkyd melamine) medium.
The resulting paints were then examined and, within practical limits, no differences in application properties were observed.

5 Example 28

250 parts (by weight) of CI Pigment Red 144 are charged to the container (See Figure). The pigment powder is then fluidised with hot air ($100^{\circ}\text{C} \pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 200 parts (by weight) of binder of composition:

- 10 1.3% Aldacol at 100% (Polyvinyl pyrrolidone)
 8.8% Reomol DX9P (Dioctyl phthalate)
 3.8% Lissapol NX (Ethoxylated nonyl phenol)
 86.1% Water

are then sprayed onto the fluidised bed of pigment uniformly over a period of 1 hour. Fluidisation is continued for a further 5 minutes. The resulting granules, of composition (by weight):

- 15 90.1% CI Pigment Red 144
 0.9% Aldacol at 100%
 6.3% Reomol DX9P
 2.7% Lissapol NX

had a Dust Index 5 (original powder 6) and a size distribution similar to that obtained in Example 1.

- 20 The granules and Original powder were then compared by incorporating equal amounts of pigment into plasticised PVC.
The resulting PVC hides were then examined and, within practical limits, no differences in application properties were observed.

Example 29

- 25 600 parts (by weight) of CI Pigment Red 101 are charged to the container (See Figure). The pigment powder is then fluidised with hot air ($100^{\circ}\text{C} \pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 100 parts (by weight) of binder of composition:

- 8.4% Vinapol 1640 at 100% (Potassium salt of polymethacrylic acid)
91.6% Water

- 30 are then sprayed onto the fluidised bed of pigment uniformly over a period of 1/2 hour. Fluidisation is continued for a further 10 minutes. The resulting granules, of composition (by weight):

- 98.3% CI Pigment Red 101
1.7% Vinapol 1640 at 100%

had a Dust Index of ~1 (Original powder ~1) and a size distribution similar to that obtained in Example 1.

- 35 1. The granules and Original powder were then compared by incorporating equal amounts of pigment into a decorative (alkyd) paint medium.
The resulting paints were then examined and, within practical limits, no difference in application properties were observed.

40 Example 30

400 parts (by weight) of CI Pigment Red 104 are charged to the container (See Figure). The pigment powder is then fluidised with hot air ($100^{\circ}\text{C} \pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 150 parts (by weight) of binder of composition:

- 45 0.8% Klucel M
 3.3% Pigment Disperser N at 100% (Sodium polyacrylate)
 12.6% Dresinol 215 at 100% (partially Dimerised rosin)
 83.4% Water

are then sprayed onto the fluidised bed of pigment uniformly over a period of 3/4 hour. Fluidisation is continued for a further 5 minutes. The resulting granules, of composition (by weight):

- 50 94.1% CI Pigment Red 104
 0.3% Klucel M
 1.2% Pigment disperser N at 100%
 4.4% Dresinol 215 at 100%

had a Dust Index of ~1 (Original powder ~2) and a size distribution similar to that obtained in Example 1.

55

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The granules and Original powder were then compared by incorporating equal amounts of pigment into a decorative (alkyd) paint medium.

The resulting paints were then examined and, within practical limits, no differences in application properties were observed.

5 Example 31

500 parts (by weight) of CI Pigment Red 101 are charged to the container (See Figure). The pigment powder is then fluidised with hot air ($100^{\circ}\text{C} \pm 10^{\circ}\text{C}$) and the outlet temperature allowed to rise to approx. 30°C . 150 parts (by weight) of binder of composition:

- 10 0.8% Klucel M (Hydroxy propyl cellulose)
 3.3% Pigment disperser N at 100% (Sodium polyacrylate)
 12.5% Dresinol 215 at 100% (partially Dimerised resin)
 83.4% Water

are then sprayed onto the fluidised bed of pigment uniformly over a period of 1/2 hour. Fluidisation is continued for a further 5 minutes. The resulting granules, of composition (by weight):

- 15 95.2% CI Pigment Red 101
 0.2% Klucel M
 3.6% Dresinol 215 at 100%
 1.0% Pigment dispersion N at 100%

had a Dust Index of ~0 (Original powder ~1) and a size distribution similar to that obtained in Example 3.

- 20 The granules and Original powder were then compared by incorporating equal amounts of pigment into a decorative (alkyd) paint medium.
 The resulting paints were then examined and, within practical limits, no differences in application properties were observed.

25 Claims

1. A process for producing a substantially dry, low dusting, free flowing granular pigment composition, comprising (a) contacting a fluidised bed of pigment, an additive system (as hereinbefore defined) and water; (b) optionally contacting the granules so obtained with the additive system defined in our copending British patent Application 39882/78; and (c) removing the granules so obtained.
- 30 2. A process as claimed in claim 1 wherein the additive system is mixed with the fluidised bed of pigment powder and sprayed with water.
 3. A process as claimed in claim 1 wherein the additive system and water are sprayed on to the fluidised bed of pigment.
 4. A process as claimed in any of the preceding claims wherein the pigment powder starting material is that produced in a conventional dry grinding or sieving process.
- 35 5. A process as claimed in any of claims 1 to 3 wherein the pigment powder starting material is that produced in dryers which give pigment directly in powder form.
 6. A process as claimed in any of the preceding claims wherein the additive system comprises a surface-active agent of the non-ionic, cationic or anionic type.
- 40 7. A process as claimed in claim 6 wherein the surfactant component of the additive system is used alone, as a mixture of different surfactants, or is used in conjunction with, or replaced by, a binder and/or applicational agent known to improve the properties of a pigment.
 8. A process as claimed in claim 7 wherein the binder is an anionic or nonionic water-soluble polymeric agent.
- 45 9. A process as claimed in claim 8 wherein the polymeric agent is hydroethyl cellulose, hydroxypropyl cellulose, sodium carboxymethyl cellulose polyvinyl alcohol or polyvinyl pyrrolidone.
 10. A process as claimed in claim 7 wherein the applicational agent is abietic acid or an ester thereof—polyethylene glycol 4000; diglycol monostearate; cellulose acetobutyrate; an alkaline earth metal salt of a fatty acid containing 12 to 20 carbon atoms; a fatty acid containing 12 to 20 carbon atoms; a fatty alcohol; an amine containing 12 to 20 carbon atoms; a vinyl chloride polymer; a vinyl chloride/vinyl acetate copolymer; polyethylene; a polyacrylonitrile or polyterpene resin; dioctyl phthalate; dicyclohexyl phthalate; a triglyceride of 12-hydroxystearic acid; a maleic or phenolic modified resin; a wood rosin modified by hydrogenation or polymerisation; or a glyceryl ester of a modified resin.
- 50 11. A process as claimed in any of the preceding claims wherein the amount of the additive system is within the range of from 0.5 to 20% by weight, based on the weight of pigment.
 12. A process as claimed in any of the preceding claims wherein the liquid used to form the solution, emulsion or dispersion of the additive system is water.
- 55

13. A process as claimed in claim 12 wherein the concentration of the additive system is within the range of from 0.5% to 60% by weight.

14. A process for producing a substantially dry, low dusting, free-flowing granular pigment composition as claimed in claim 1 substantially as described with reference to any of the examples.

5 15. A substantially dry, low-dusting, free-flowing granular pigment composition when produced by a process claimed in any of the preceding claims. 5

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B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C09B

Documentation searched (other than minimum documentation) to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP 1 195 411 A (CLARIANT GMBH) 10 April 2002 (2002-04-10) cited in the application claims 1-15	1-14
Y	EP 1 195 414 A (CLARIANT GMBH) 10 April 2002 (2002-04-10) cited in the application claims 1-12	1-14
Y	EP 1 195 413 A (CLARIANT GMBH) 10 April 2002 (2002-04-10) cited in the application claims 1-16	1-14
A	US 6 340 387 B1 (J. ORTH-GERBER ET AL.) 22 January 2002 (2002-01-22) claims 1-11	1-14
-/-		

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Date of the actual completion of the international search

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No
A	US 3 607 336 A (E. E. JAFFE) 21 September 1971 (1971-09-21) claims 1-7	1-14
A	GB 2 009 204 A (CIBA-GEIGY AG) 13 June 1979 (1979-06-13) claims 1-15	1-14
A	PROCEEDINGS MICRO. TEC. VDE WORLD MICROTECHNOLOGIES CONGRESS, PROCEEDINGS OF INTERNATIONAL CONFERENCE ON MICROTECHNOLOGIES, vol. 1, 2000, pages 401-405, XP0001058349 * entire document *	1-14

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No.

PCT/EP 03/10610

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
EP 1195411	A	10-04-2002	DE 10049200 A1	11-04-2002
			EP 1195411 A1	10-04-2002
			JP 2002129050 A	09-05-2002
			US 2002055619 A1	09-05-2002
EP 1195414	A	10-04-2002	DE 10049202 A1	11-04-2002
			EP 1195414 A1	10-04-2002
			JP 2002161218 A	04-06-2002
			US 2002040662 A1	11-04-2002
EP 1195413	A	10-04-2002	DE 10049201 A1	11-04-2002
			EP 1195413 A1	10-04-2002
			JP 2002155221 A	28-05-2002
			US 2002040665 A1	11-04-2002
US 6340387	B1	22-01-2002	DE 19910521 A1	28-09-2000
			AU 4284400 A	28-09-2000
			BR 0008835 A	18-12-2001
			CA 2362443 A1	14-09-2000
			CZ 20012103 A3	12-09-2001
			WO 0053679 A1	14-09-2000
			EP 1165697 A1	02-01-2002
			JP 2002538279 A	12-11-2002
			PL 350581 A1	13-01-2003
US 3607336	A	21-09-1971	NONE	
GB 2009204	A	13-06-1979	CA 1122784 A1	04-05-1982
			CH 635862 A5	29-04-1983
			DE 2844710 A1	26-04-1979
			DK 457278 A , B,	16-04-1979
			FR 2405983 A1	11-05-1979
			IT 1159934 B	04-03-1987
			JP 1430547 C	24-03-1988
			JP 54065731 A	26-05-1979
			JP 62039181 B	21-08-1987
			NL 7810333 A	18-04-1979
			US 4264552 A	28-04-1981

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